JAPANESE [JP,09-071417,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION TECHNICAL PROBLEM MEANS WRITTEN AMENDMENT

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention has high ultraviolet rays screening ability, and its transparency over visible light is high, it is uniform, is excellent in a using feeling, and relates to the cosmetics which blended the flake-like granular material and it which do not almost have alkaline elution.
[0002]
[Description of the Prior Art]Paints class titanium oxide with large (200-300 nm) primary particle diameter and particulate titanium oxide with small (10-50 nm) primary particle diameter are among the titanium oxide blended with cosmetics. Obliterating power was large, and when it

applied to the skin, the former changed into the so-called state of a thick makeup, became an unnatural result, and since the adhesion force to skin was large, it had the tendency for a smooth feel and extensibility (mileage) to be insufficient.

[0003]It was difficult for the latter to carry out uniform dispersion to a medium, and even if it made it distribute once, there was a problem which condenses temporally, forms lumps or becomes nonuniformity. Especially when abundant combination was carried out as cosmetics, the above-mentioned problem became remarkable, and since the adhesion force to skin was still larger, there was a problem that a smooth feel and extensibility (mileage) were insufficient.

[0004]In order to solve the above-mentioned problem, on the surface of mica with a mean particle diameter of 0.5-100 micrometers. The ultraviolet-rays cover paints which cover a particulate titanium dioxide in the covering thickness which is 5 to 20% of the weight of coating volume, and a feeling of pearly luster, brightness, and obliterating power do not reveal on parenchyma to mica are proposed (JP,5-87545,B). When these ultraviolet-rays cover paints are used as cosmetics, it is uniform and a smooth feel and extensibility (mileage) are acquired, but with an insufficient transparent feeling. Although it had the cleavage which requires cost for arranging the thickness which may be colored, alkali was eluted from the mica from which a cleavage may not arise thoroughly in the surface, but thickness may change stair-like in the mica flake of one sheet, and there were new problems -- it may have an adverse effect on the skin.

[0005]On the other hand, after this invention persons apply on a substrate desirable and smooth in the surface, dry and make the solution containing an organic metallic compound exfoliate from a substrate on a substrate, they have proposed heat-treating flake-like glass and a manufacturing method for the same. (For example, JP,3-285838,A, JP,4-37622,A). The various flake-like glass manufactured in accordance with this method is excellent in surface smoothness, its thickness is also uniform, and the slide of mileage is well good. The pearly luster pigment which covered a titania, zirconia, or those mixtures to the these flake-like glass surface is also proposed. (JP,6-116507,A). Since a substrate

is synthetic glass which contains silica not less than 80%, these paints have high transparency and are colorless. It is uniform thickness, and surface smoothness is high, there is also no alkaline elution and safety is high. However, there is no smooth feel depending on the thickness and the aspect ratio of a substrate, and there was a difficulty said that mileage is also bad.

[0006]

[Problem(s) to be Solved by the Invention]In view of the above-mentioned conventional technology, this invention has the high ultraviolet rays screening ability which was not manufactured conventionally, and its transparency over visible light is high, is uniform, is excellent in a using feeling, and provides the quality cosmetics which blended the flake-like granular material and it which do not almost have alkaline elution.

[0007]

[Means for Solving the Problem]In order to solve this technical problem, this invention persons have added examination about a use feel as thickness, particle diameter, and cosmetics of a flake-like metallic oxide which is the substrate about a flake-like metallic oxide which covered a titanium oxide layer. As a result, thickness of a flake-like metallic oxide was 0.1 micrometers or more 1 micrometer or less, and when it used as cosmetics that aspect ratios are 5-150, it is uniform, found out that a smooth feel and extensibility (mileage) were acquired, and resulted in this invention.

[0008]This invention to namely, a metallic-oxide flake-like powder surface which uses as the main ingredients amorphous silica whose average thickness is 0.1-1 micrometer, and whose aspect ratios are 5-100. They are the cosmetics which blended a titanium oxide covering metal oxide flake-like granular material and it which covered a 5-400-nm-thick titanium oxide layer.

[0009]A metallic-oxide flake-like granular material used in this invention is producible from liquid containing an organic metallic compound in which hydrolysis and a polycondensation are possible by a method (for example, JP,3-285838,A, JP,4-37622,A) which this invention persons already proposed.

[0010]That is, apply liquid containing an organic metallic compound in which hydrolysis and a polycondensation are

possible on a substrate and a desirable substrate smooth in the surface, and dry this, a gel film is made to exfoliate from a substrate, it sinters, and a flake-like metallic oxide is produced.

[0011]As an organic metallic compound in which hydrolysis and condensation polymerization as a raw material used for this invention are possible, a metal alkoxide which has an alkoxyl group is preferred. Specifically, methoxides, such as silicon, titanium, aluminum, and a zirconium, ethoxide, propoxide, butoxide, etc. are used as a simple substance or a mixture. A presentation of a flake-like granular material obtained At least 70% of the weight of amorphous silica [70 to 100 % of the weight of], Since it is a metallic oxide which consists of at least one sort of a total of 0 to 30 % of the weight chosen from alumina, a titania, and zirconia and softening temperature (temperature in case coefficient of viscosity is 7th power POISE of 4.5x10) is not less than 1100 **, temperature at the time of covering and heat-treating titanium oxide can be borne.

[0012]In this invention, average thickness of the above-mentioned metallic-oxide flake-like granular material is 0.1-1 micrometer, and aspect ratios are 5-150. A titanium oxide coating of a flake-like metallic oxide which fulfills this condition is uniform when it is used as cosmetics, and a smooth feel and extensibility (mileage) are acquired. Preferably, average thickness is 0.3-0.9 micrometer, an aspect ratio is [10-100, and mean particle diameter] 5-90 micrometers, and a smooth feel and extensibility (mileage) of a titanium oxide covering metal oxide flake-like granular material are better. Still more preferably, average thickness is 0.4-0.8 micrometer, an aspect ratio is [20-80 and mean particle diameter] 15-50 micrometers, and a smooth feel and extensibility (mileage) of a titanium oxide covering metal oxide flake-like granular material are dramatically good.

[0013]As for a method of covering titanium oxide to a metallic-oxide flake-like granular material, a known method is used. For example, generally a method (for example, JP,43-25644,B) of hydrolyzing sulfuric acid oxy titanium sulfate by boiling temperature and a hydrolysis method (for example, JP,49-3824,B) of titanium tetrachloride are known.

[0014]After covering a titanium oxide precursor with these methods, it heat-treats at 700-1000 **, and makes by them

with a titanium oxide enveloping layer whose density it is stable and is large.

[0015]Covering thickness of the above-mentioned titanium oxide layer in this invention is 5-400 nm. If thickness is thinner than 5 nm, sufficient ultraviolet-rays shielding effect is not accepted and it is not desirable. On the other hand, since physical properties in particular, such as an ultraviolet-rays shielding effect and an interference color, do not change even if thickness is thicker than 400 nm, it becomes waste of titanium oxide. Preferably, titanium oxide layer thickness is 20-160 nm.

[0016]When the above-mentioned titanium oxide layer thickness is smaller than 40 nm, a titanium oxide covering metal oxide flake-like granular material is the high white of transparency. When titanium oxide layer thickness is 40-60 nm, as for a titanium oxide covering flake-like metallic oxide, high coloring (interference color) of transparency which has yellow, red, a red purple color, and a blue and green transparent feeling when it becomes silver and titanium oxide layer thickness is further increased one by one to 160 nm is accepted. If titanium oxide layer thickness is furthermore increased, golden color, red, a red purple color, and the same blue and green color will be accepted. Therefore, coloring with a general transparent feeling is accepted with titanium oxide layer thickness up to 160 nm.

[0017]A little unlike it of titanium oxide covering mica in which these coloring is generally marketed, a transparent feeling is accepted more. It is mentioned that a metallic-oxide flake-like granular material which is a substrate has high transparency as the reason, having high surface smoothness, that it is amorphous (glass), etc.

[0018]When using it as cosmetics, these transparent feelings and the coloring can improve design nature, and are used preferably.

[0019]Light transparency is high, and cannot serve as an irregular color easily, and cosmetics which blended a titanium oxide covering metal oxide flake-like granular material of this invention serve as a color-enhancing good stable product. Ultraviolet-rays shielding efficiency is high and high ultraviolet-rays cover is possible at a little combination. Since a titanium oxide covering metal oxide flake-like granular material shows good slide nature, without condensing mutually, extensibility (mileage) is

good and serves as a product excellent in use tactile feeling. Since impurity quantity of a metallic-oxide flake-like granular material can be stopped low, there is also almost no alkaline elution which is observed in mica, and it becomes a safe quality product to the skin.

[0020]Even if it uses together paints etc. which are usually used if needed besides the above-mentioned titanium oxide covering metal oxide flake-like granular material, it does not interfere with cosmetics said by this invention at all. For example, titanium oxide, a zinc oxide, zirconium oxide, Synthetic Ochre, black iron oxide, Inorganic pigments, such as rouge, ultramarine, Prussian blue, chrome oxide, and chromium hydroxide, a mica titan, Pearly luster pigments, such as bismuth oxychloride, tar dye, natural coloring matter, silica beads, Granular materials, such as plastic beads, such as nylon and an acrylic, talc, kaolin, mica, a sericite, other mica, magnesium carbonate, calcium carbonate, aluminum silicate, a magnesium silicate, and clay are illustrated.

[0021]Although it changes with kinds of cosmetics made into the purpose as loadings of the above-mentioned titanium oxide covering metal oxide flake-like granular material, it is used in 1 to 80% of the weight of the range to solid ingredients, such as paints, and 2 to 50% of the weight of a range is especially preferred. In content not more than this, even if there is a difficulty that coloring by which an ultraviolet-rays shielding effect is not demonstrated notably is not good etc. and it adds many metallic-oxide flake-like granular materials from a maximum conversely, an ultraviolet-rays shielding effect does not go up, but other pigment components decrease and it becomes difficult to prepare a color tone or to raise adhesion to the skin.

[0022]In order to raise dispersibility in inside of cosmetics of a titanium oxide covering metal oxide flake-like granular material used by this invention or to improve a feel, performing and reforming a surface treatment of a titanium oxide covering metal oxide flake-like granular material does not interfere at all. For example, methil hydrogen polysiloxane, a reactant alkyl polysiloxane, If a surface treatment is performed by what is called hydrophobing agents, such as metal salt chosen from aluminum of hydrogenation lecithin, acylamino acid, and acylation collagen besides metallic soap, magnesium, calcium,

titanium, zinc, a zirconium, and iron, Since the surface of flake-like glass changes from hydrophilic nature to hydrophobicity, familiarity by oils added at the time of preparation of cosmetics becomes good, and it serves as good cosmetics of a feel.

[0023]

[Embodiment of the Invention]An example is shown below. An example-1 silicontetramethoxide, ethanol, and water were mixed at a rate of 1:2:1 by the volume ratio, and stirring was performed at 50 ** for 15 hours. Pulled up the liquid previously prepared on the 1-mm-thick stainless plate at 20 cm x 20 cm which ground the surface with the dip coating method, formed membranes by a part for 30-cm/in speed, it was made to dry for 1 minute within 150 ** oven, and the silica gel membrane which exfoliated from the stainless plate was collected with the nylon brush. Collected flake-like silica gel was heat-treated at 1000 ** for 2 hours, and flake-like silica glass was obtained.

[0024]When flake-like silica glass was observed with the scanning electron microscope, it was dramatically smooth in the surface and thickness was about 0.6 micrometer.

[0025]The above-mentioned flake-like silica glass was ground and classified with the jet mill, and it was considered as the mean particle diameter of about 30 micrometers. The aspect ratio of the classification flake-like silica glass at this time is about 50.

[0026]100 ml of water was made to distribute this classification flake-like silica glass 15g, and it kept at 80 **. Oxy titanium sulfate solution [equivalent to a 4-g titanium dioxide / 100g and 50g] 50% sulfuric acid was added here slowly. After carrying out about 1-hour and half heating boil of this, the slurry was filtered, it washed with water, sulfuric acid was removed, and it dried at 120 ** for 2 hours.

[0027]The obtained titanium oxide precursor covering flake-like silica glass was heat-treated at 1000 ** for 1 hour, and the high white powder (titanium oxide covering flake-like silica glass) of the transparent feeling was obtained. When observed with the scanning electron microscope, the thickness of the titania layer was about 25 nm. The thickness, the mean particle diameter, and the aspect ratios of titanium oxide covering flake-like silica glass are about 0.6 micrometer, about 30 micrometers, and about 50, respectively, and were not different from it of the

classification flake-like silica glass before covering titanium oxide.

[0028]This titanium oxide covering flake-like silica glass is distributed about 10% of the weight in vinyl system resin (the refractive index after hardening is about 1.5), The place which measured transmissivity with the spectrophotometer as a film of about 0.15-mm thickness, Visible light transmittance with a wavelength of 600-800 nm was not less than 80% over the whole region, and ultraviolet ray transmission with a wavelength of 300-350 nm was 0 to 5%, the transparency over visible light was high and covering ultraviolet rays effectively was checked.

[0029]After adding the water 50g which was freshly boiled to the above-mentioned titanium oxide covering flake-like silica glass 5g, and was cooled to it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.5 and has checked that it was neutrality mostly.

[0030]When a little above-mentioned titanium oxide covering flake-like silica glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, it was dramatically smooth and excelled in mileage.

[0031]The unground flake-like silica glass (about 0.6 micrometer in thickness) produced by the method of the comparative example-1 example-1 statement was ground and classified with the jet mill, and it was considered as the mean particle diameter of about 2 micrometers. The aspect ratio of the flake-like silica glass at this time is 3.3.

[0032]100 ml of water was made to distribute this classification flake-like silica glass 15g, and it kept at 80 **. Oxy titanium sulfate solution [equivalent to an 8-g titanium dioxide / 100g and 50g] 50% sulfuric acid was added here slowly. After carrying out about 1-hour and half heating boil of this, the slurry was filtered, it washed with water, sulfuric acid was removed, and it dried at 120 ** for 2 hours.

[0033]The obtained titanium oxide precursor covering flake-like silica glass was heat-treated at 1000 ** for 1 hour, and the high white powder (titanium oxide covering flake-like silica glass) of the transparent feeling was obtained. When observed with the scanning electron microscope, the thickness of the titania layer was about 25 nm.

[0034]This titanium oxide covering flake-like silica glass is

distributed about 10% of the weight in vinyl system resin (the refractive index after hardening is about 1.5), As a film of about 0.15-mm thickness, when transmissivity was measured with the spectrophotometer, visible light transmittance with a wavelength of 600-800 nm was 50 to 70% over the whole region, and ultraviolet ray transmission with a wavelength of 300-350 nm was 0 to 5%. Since the flake was [the transparency over visible light] small, although it was not so high, covering ultraviolet rays effectively was checked.

[0035]After adding the water 50g which was freshly boiled to the above-mentioned titanium oxide covering flake-like silica glass 5g, and was cooled to it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.5 and has checked that it was neutrality mostly.

[0036]When a little above-mentioned titanium oxide covering flake-like silica glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, the slide was bad and the feel was [the bottom] bad coarsely at sensibility.

[0037]Powder foundation was produced by example-2, comparative example-2, and combination not more than comparative example-3.

[0038]

Ingredient-1 Loadings (% of the weight)
---------- flake of this invention produced by example-1 14.1 talc . 74.7 titanium oxide (primary particle diameter of 200-250 nm) 3.8 particulate titanium oxide (primary particle diameter of 30-50 nm) 1.9 magnesium stearates 2.9 rouge 0.5 Synthetic Ochre 0.8 black iron oxide 0.1 silk powder 0.5[0039]

Ingredient-2 Loadings (% of the weight)
---------- Squalane 0.5 sorbitan sesquioleate 0.1[0040]

Ingredient-3 Loadings (% of the weight)
---------- Perfume 0.1[0041]Ingredient-1 was agitated for 5 minutes using the Henschel mixer. Agitation mixing was performed while the ingredient uniformly fused at 70 ** was dropped at this. After adding ingredient-3, agitation mixing was carried out for 1 minute, the atomizer ground, and product-1 (example-2) was obtained.

[0042]Titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1 (0.6 micrometer in thickness of a substrate.) Product-2 (comparative example-2) was obtained by the completely same method as the above except having added the titanium oxide covering flake-like silica glass (0.6 micrometer in thickness of a substrate, the particle diameter of 2 micrometers, aspect ratio 3.3) produced by comparative example-1 instead of the particle diameter of 30 micrometers, and the aspect ratio 50.
[0043]Product-3 (comparative example-3) was obtained by the completely same method as the above except not having added the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1.
[0044]After [product-1, product-2, and product-3] having extracted 0.22 g, respectively, applying to 4 cm x 5 cm of skins of five test subjects' back center section adjacently uniformly and exposing to direct sunlight between 11:00 a. m. and 2:00 p.m. in Izu in August for 30 minutes - 3 hours, the suntan extent was observed.
[0045]Table-1 shows the result. The average value of the grade of five test subjects' suntan was judged by the following classification.
1; -- 3; having hardly got tanned but having done 2; suntan of turns out to be -- 4; which got tanned a little red -- 5; which got tanned quite red -- intense -- it got tanned and the skin exfoliated behind [0046]
[Table 1]
Table -One ==========
Granular material of this invention Comparative granular material Comparative granular material (product-1) (product-2) (product-3)
(Example-2) (comparative example-2) (comparative example-3)
---------- After 30 minutes 11 1 1 hour after 1 1 3 2 hours after 1 1 4 3 hours after 2 2 5
========== [0047]
Although a difference was hardly visually accepted after 30-minute progress, A remarkable difference comes to be accepted after 1-hour progress, Suntan of the skin which applied the granular material (product-2, comparative example-2) which blended the titanium oxide covering flake-like silica glass produced by granular material (product-1,

example-2) and comparative example-1 which blended the titanium oxide covering flake-like silica glass of this invention, As compared with the case where the granular material (product-3, comparative example-3) which did not blend the above-mentioned titanium oxide covering flake-like silica glass is applied, it decreased substantially.
[0048]20 female panelists are made to use the three above-mentioned sorts of products for ten days, and the result of the sensory test which evaluated the peak by five step-by-step procedures of five points and 1-5 points to carry out is shown in table-2.
[0049]
[Table 2]
Table -Two ==========
Granular material of this invention Comparative granular material Comparative granular material (product-1) (product-2) (product-3)
(Example-2) (comparative example-2) (comparative example-3)
---------- Mileage 4.8 1.5 With 1.8 4.5 3.0 3.8 transparent feelings 4.7 2.0 3.4 glossy senses 4.6 2.0 2.6 sense of color 4.2 2.8 3.7 performance durability 4.8 3.1 2.0 ========== [0050] thus -- the cosmetics of this invention are extended -- being all out (adhesion) -- it was good, the transparent feeling and the glossy sense were good, and it was checked that it excels in coloring and is [it fades and] hard to make it it.
[0051]Example-3 silicontetraethoxide, ethanol, and decinormal chloride were mixed at a rate of 10:20:1 by the volume ratio, and stirring was performed at 40 ** for 2 hours. Next, titanium isopropoxide was added so that it might contain 10% of the weight as an oxide after sintering, and stirring was continued. Added the water of 45 volume % of this liquid, and it was made to react at 40 ** for 20 hours, and was considered as coating liquid.
[0052]The surface at ground 20 cm x 20 cm on a 1-mm-thick stainless plate, Pulled up the coating liquid prepared previously with the dip coating method, formed membranes by a part for 45-cm/in speed, it was made to dry for 1 minute within 150 ** oven, and the silica gel membrane which exfoliated from the stainless plate was collected with the nylon brush. The collected flake-like gels were heat-treated at 1000 ** for 2 hours, and flake-like silica titania

glass was obtained. The content of the titania was 9.8 % of the weight as a result of analysis.

[0053]When flake-like silica titania glass was observed with the scanning electron microscope, it was dramatically smooth in the surface and thickness was about 0.85 micrometer.

[0054]The above-mentioned flake-like silica titania glass was ground and classified with the ball mill, and it was considered as the mean particle diameter of about 80 micrometers. The aspect ratio of the classification flake-like silica titania glass at this time is about 94.

[0055]100 ml of water was made to distribute this classification flake-like silica titania glass 15g, and it kept at 80 **. Oxy titanium sulfate solution [equivalent to a 21-g titanium dioxide / 150g and 50g] 50% sulfuric acid was added here slowly. After carrying out about 1-hour and half heating boil of this, the slurry was filtered, it washed with water, sulfuric acid was removed, and it dried at 120 ** for 2 hours.

[0056]The obtained titanium oxide precursor covering flake-like silica titania glass was heat-treated at 1000 ** for 1 hour, and the high red granular material (titanium oxide covering flake-like silica titania glass) of the transparent feeling was obtained. When observed with the scanning electron microscope, the thickness of the titania layer was about 90 nm.

[0057]This titanium oxide covering flake-like silica titania glass is distributed about 10% of the weight in vinyl system resin (the refractive index after hardening is about 1.5), The place which measured transmissivity with the spectrophotometer as a film of about 0.15-mm thickness, Visible light transmittance with a wavelength of 600-800 nm was 70 to 85% over the whole region, and ultraviolet ray transmission with a wavelength of 300-350 nm was 0 to 5%, the transparency over visible light was high and covering ultraviolet rays effectively was checked.

[0058]After adding the water 50g which was freshly boiled on the above-mentioned titanium oxide covering flake-like silica titania glass 5g, and was cooled on it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.4 and has checked that it was neutrality mostly.

[0059]When a little above-mentioned titanium oxide

covering flake-like silica glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, it was quite smooth and mileage was good.
[0060]The unground flake-like silica titania glass (about 0.85 micrometer in thickness) produced by the method of the comparative example-4 example-3 statement was ground and classified with the ball mill, and it was considered as the mean particle diameter of about 200 micrometers. The aspect ratio of the flake-like silica glass at this time is about 235.
[0061]On this classification flake-like silica titania glass, the titanium oxide layer of about 90-nm thickness was covered with the same method as example-3. The transparency and ultraviolet rays screening ability of this titanium oxide covering flake-like silica titania glass were almost the same as the titanium oxide covering flake-like silica titania glass of example-3 statement.
[0062]After adding the water 50g which was freshly boiled on the above-mentioned titanium oxide covering flake-like silica titania glass 5g, and was cooled on it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.5 and has checked that it was neutrality mostly.
[0063]However, when a little above-mentioned titanium oxide covering flake-like silica titania glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, he had sensibility caught on skin, the slide was bad, and a feel was not so good.
[0064]Using the liquid prepared by the method of the comparative example-5 example-3 statement, raising speed of the stainless steel board was considered as a part for 60-cm/more greatly than example-3, and flake-like silica titania glass was produced by the method of the example-3 statement. When thickness was measured with the scanning electron microscope, it was about 1.5 micrometers.
[0065]This flake-like silica titania glass was ground and classified with the ball mill, and it was considered as the mean particle diameter of about 80 micrometers. The aspect ratio of this classification flake-like silica titania glass is about 53.
[0066]On this classification flake-like silica titania glass, the titanium oxide layer of about 90-nm thickness was covered with the same method as example-3. The transparency and

ultraviolet rays screening ability of this titanium oxide covering flake-like silica titania glass were almost the same as the titanium oxide covering flake-like silica titania glass of example-3 statement.

[0067]After adding the water 50g which was freshly boiled on the above-mentioned titanium oxide covering flake-like silica titania glass 5g, and was cooled on it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.5 and has checked that it was neutrality mostly.

[0068]However, when a little above-mentioned titanium oxide covering flake-like silica titania glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, he had rough sensibility, mileage was bad, and a feel was not so good.

[0069]Powder foundation was produced in example-4, comparative example-6, and the procedure not more than comparative example-7.

[0070]Instead of the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1 in example-2, Product-4 (example-4) was obtained by the completely same method as example-2 except having added the titanium oxide covering flake-like silica titania glass (0.85 micrometer in thickness of a substrate, the particle diameter of 80 micrometers, aspect ratio 94) produced by example-3.

[0071]Instead of the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1, Product-5 (comparative example-6) was obtained by the completely same method as example-2 except having added the titanium oxide covering flake-like silica titania glass (0.85 micrometer in thickness of a substrate, the particle diameter of 200 micrometers, aspect ratio 235) produced by comparative example-4.

[0072]Instead of the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1, Product-6 (comparative example-7) was obtained by the completely same method as example-2 except having added the titanium oxide covering flake-like silica titania glass (1.5 micrometers in thickness of a substrate, the particle diameter of 80 micrometers, aspect ratio 53) produced by comparative example-5.

[0073]20 female panelists are made to use the three above-

mentioned sorts of products for ten days, and the result of the sensory test which evaluated the peak by five step-by-step procedures of five points and 1-5 points to carry out is shown in table-3.

[0074]

[Table 3]

Table -Three

========== Granular material of this invention Comparative granular material Comparative granular material (product-4) (product-5) (product-6)

(Example-4) (comparative example-6) (comparative example-7)

---------- Mileage 4.6 3.2 With 2.8 4.5 2.8 3.8 transparent feelings 4.7 4.2 4.2 glossy senses 4.6 4.0 3.8 sense of color 4.1 3.8 3.7 performance durability 4.7 4.0 4.0 ========== [0075] thus -- the cosmetics of this invention are extended -- being all out (adhesion) -- it was good, the transparent feeling and the glossy sense were good, and it was checked that it excels in coloring and is [it fades and] hard to make it it.

[0076]100 ml of water was made to distribute 15 g of flake-like silica glass (0.6 micrometer in thickness, the particle diameter of 30 micrometers, aspect ratio 50) which ground [which ground and produced] and classified by the method of the example-5 example-1 statement, and it kept at 80 **. Oxy titanium sulfate solution [equivalent to an 11-g titanium dioxide / 100g and 50g] 50% sulfuric acid was added here slowly. After carrying out about 1-hour and half heating boil of this, the slurry was filtered, it washed with water, sulfuric acid was removed, and it dried at 120 ** for 2 hours.

[0077]The obtained titanium oxide precursor covering flake-like silica glass was heat-treated at 1000 ** for 1 hour, and the high yellow granular material (titanium oxide covering flake-like silica glass) of the transparent feeling was obtained. When observed with the scanning electron microscope, the thickness of the titania layer was about 65 nm.

[0078]This titanium oxide covering flake-like silica glass is distributed about 10% of the weight in vinyl system resin (the refractive index after hardening is about 1.5), The place which measured transmissivity with the spectrophotometer

as a film of about 0.15-mm thickness, Visible light transmittance with a wavelength of 600-800 nm was 75 to 90% over the whole region, and ultraviolet ray transmission with a wavelength of 300-350 nm was 0 to 5%, the transparency over visible light was high and covering ultraviolet rays effectively was checked.

[0079]After adding the water 50g which was freshly boiled on the above-mentioned titanium oxide covering flake-like silica titania glass 5g, and was cooled on it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 6.2 and has checked that it was neutrality mostly.

[0080]When a little above-mentioned titanium oxide covering flake-like silica glass was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, it was dramatically smooth and excelled in mileage.

[0081]In comparative example-8 example-5, it is flake-like silica glass (0.6 micrometer in thickness.). The white mica (0.6 micrometer in thickness, the particle diameter of 30 micrometers, aspect ratio 50) which ground and classified was used instead of the particle diameter of 30 micrometers, and the aspect ratio 50, and also it is the same method, and 65-nm-thick titanium oxide was covered. Although the obtained titanium oxide covering white mica was glossy yellow, the transparent feeling was seldom sensed.

[0082]This titanium oxide covering white mica is distributed about 10% of the weight in vinyl system resin (the refractive index after hardening is about 1.5), As a film of about 0.15-mm thickness, when transmissivity was measured with the spectrophotometer, visible light transmittance with a wavelength of 600-800 nm was 45 to 65% over the whole region, and ultraviolet ray transmission with a wavelength of 300-350 nm was 0 to 5%. Although ultraviolet rays were covered effectively, it was checked that the transparency over visible light is not so good.

[0083]After adding the water 50g which was freshly boiled to the above-mentioned titanium oxide covering white mica 5g, and was cooled to it and stirring it with for [sufficient] 3 minutes, when the pH of the filtered liquid was measured, it is 8.5 and it turned out that it is alkalinity. It can be said that it is because alkalinity was eluted from white mica.

[0084]When a small amount of above-mentioned titanium

oxide covering white mica was taken in the hand and the slide on skin, mileage, etc. were investigated in organoleptics, there is a certain amount of smoothness and the feel of what also has good mileage was a little inferior to the titanium oxide covering flake-like silica glass produced by example-5.

[0085]Powder foundation was produced in example-6 and the procedure not more than comparative example-9.

[0086]Instead of the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1 in example-2, Product-7 (example-6) was obtained by the completely same method as example-2 except having added the titanium oxide covering flake-like silica glass (0.6 micrometer in thickness of a substrate, the particle diameter of 30 micrometers, aspect ratio 50) produced by example-5.

[0087]Instead of the titanium oxide covering flake-like silica glass of this invention produced by example-1 in ingredient-1, Product-8 (comparative example-9) was obtained by the completely same method as example-2 except having added the titanium oxide covering white mica (0.6 micrometer in thickness of a substrate, the particle diameter of 30 micrometers, aspect ratio 50) produced by comparative example-8.

[0088]20 female panelists are made to use the two above-mentioned sorts of products for ten days, and the result of the sensory test which evaluated the peak by five step-by-step procedures of five points and 1-5 points to carry out is shown in table-4.

[0089]

[Table 4]

Table -Four ==========

granular material of this invention Comparative granular material (product-7) (product-8)
(Example-6) (comparative example-9)
---------- Mileage 4.8 4.6 with 3.8 4.5
transparent feelings 4.5 3.0 glossy senses 4.6 4.8 sense of color 4.1 3.5 performance durability 4.6 4.0
========== [0090]Thus, mileage was better than the conventional cosmetics to which the cosmetics of this invention used the substrate as white mica, the transparent feeling was good and excelling in sense of color was checked.

[0091]
[Effect of the Invention]By the above detailed description of the invention and the example, and a comparative example, according to this invention, it has high ultraviolet rays screening ability so that clearly, and the transparency over visible light is high, and it is uniform, and excels in a using feeling, and the quality cosmetics which blended the flake-like granular material and it which do not almost have alkaline elution are obtained.

[Translation done.]